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# PROCEDURE FOR SIEVING SOIL AND ROCK SAMPLES

***LOS ALAMOS***
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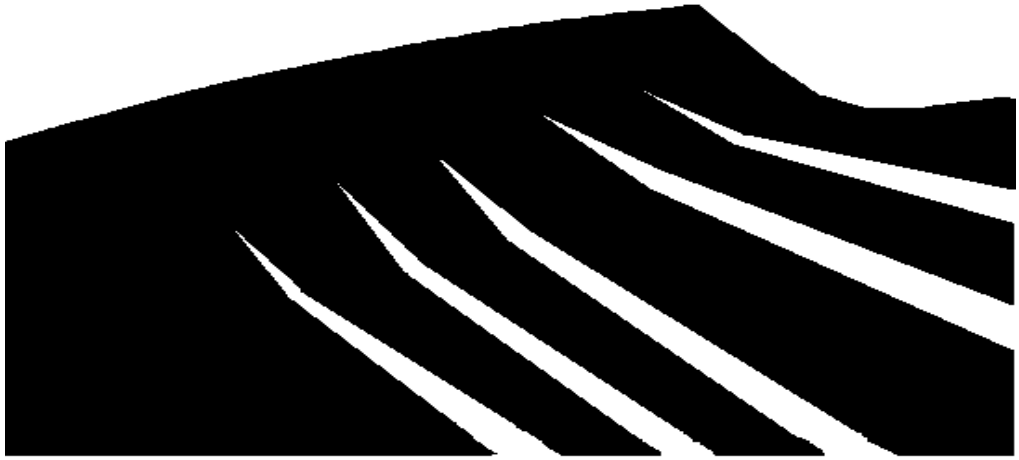

APPROVAL FOR RELEASE

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**Los Alamos**
**Yucca Mountain Site Characterization Project**

# HISTORY OF REVISION

| REVISION NO. | EFFECTIVE DATE | PAGES REVISED | REASON FOR CHANGE |
|---|---|---|---|
| R0 | 01/06/92 | N/A | Initial procedure. |
| R1 | 12/23/96 | All | Revised to comply with LANL-YMP-QP-06.3 requirements. |

# PROCEDURE FOR SIEVING SOIL AND ROCK SAMPLES

## 1.0 PURPOSE

This detailed technical procedure (DP) describes the procedure for sieving soil and rock samples prior to additional processing for moisture, chemical, isotopic or other analyses for the Yucca Mountain Site Characterization Project (YMP).

## 2.0 SCOPE

This DP applies to YMP personnel who sieve soil and rock samples for moisture, chemical, isotopic or other analyses as part of the YMP's Water Movement Test task for Los Alamos National Laboratory (LANL).

## 3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-CST-DP-90, Measurement of Moisture Content of Soil Samples
LANL-CST-DP-92, Sample Leaching to Extract Soluble Chloride and Bromide
LANL-CST-DP-103, Identification, Storage, and Handling of Samples for the Water Movement Test
LANL-CST-DP-109, Particle Size Reduction of Samples

## 4.0 DEFINITIONS

### 4.1 Soil and Rock Samples

Soil and rock samples are materials intended for laboratory studies or analyses that were obtained directly from the Yucca Mountain area or obtained from special sources such as prototype testing localities. Soil includes any unconsolidated material above bedrock, i.e., the regolith. Rock samples may include, but are not limited to, surface samples, drill cores, drill cuttings, and rocks from underground excavations.

### 4.2 Gravel-free Fraction

The gravel-free fraction of a soil sample is that portion which passes through a 2-mm sieve.

### 4.3 Rock Flour

Rock flour consists of those particles smaller than fine sand, i.e., which pass through a 0.105-mm sieve.

### 4.4 Large Fragments

Large rock fragments are those pieces which are too large to be leached efficiently following methods described in DP-92. These fragments are functionally defined as those retained by a 12.5-mm (0.5-in) sieve.

## 5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure:

- Principal Investigator (PI) for the Water Movement Test
- YMP personnel performing work to the procedure

## 6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified, replaced by a new document, or until current work practice is documented in accordance with QP-03.5, Section 6.1.6.
- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.
- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

### 6.1 Principle

A soil sample may need to be sieved if the gravel-free fraction provides a more useful basis for reporting moisture content (determined by DP-90) than does the total soil mass (section 6.5.2). The gravel-free fraction is that portion passing a 2-mm sieve.

A rock sample may need to be sieved if the sample has a significant fraction either of fine-grained material (rock flour) or of large rock fragments (section 6.5.3). Leaching rock flour in a given sample might release an excessive quantity of rock halides, thereby diluting the meteoric signal. Consequently, the PI may judge that leaching should proceed on particle sizes greater than the size of fine sand (particles retained by a 0.105-mm sieve). At the other extreme, some rock

fragments may be too large to be leached efficiently using the methods described in DP-92. In order to increase the surface area available for leaching, the PI may judge that the larger fragments (i.e., those larger than about 1.25 cm (0.5 in)), should be isolated from the sample either by sieving or hand picking. These fragments may then be reduced in size by some appropriate method (DP-109).

A standardized sieving procedure will ensure the consistency and integrity of analytical results as well as reduce the possibility of contamination by sources of chloride, bromide, or other constituents other than those in the sample being processed.

## 6.2 Equipment and Hardware/Software

Equipment needed to sieve samples is listed below. Items equivalent to those listed below may be used provided they perform the same function with an acceptable level of performance as judged by the user or the PI.

- water purification system capable of producing deionized water with resistivity of at least 17.5 megohms-cm (calibration not required, see section 6.3.2)
- analytical balance, with minimum of 160 gram capacity and 0.1 mg sensitivity (e.g., Mettler Model AE 100) (calibration not required; see section 6.3.2)
- balance with at least 1 kg capacity and 1 g sensitivity (calibration not required, see section 6.3.2)
- convection oven capable of maintaining the temperature at 105°C ± 5°C
- sieve set including mesh sizes of 0.105 mm, 2 mm, and 1.25 cm; the use of intermediate mesh sizes not listed is allowed and may increase the efficiency of sieving

### 6.2.1 Equipment Malfunctions

Any equipment malfunction occurring during implementation of this procedure is likely to be readily detectable in the course of conducting work and hence is not expected to have a detrimental effect on the final results. The water purification system has a meter that indicates the resistivity of the purified water. This meter is checked before each use to verify that the resistivity is within the range of acceptable values, i.e., greater than 17.5 megohm-cm. If a problem with any equipment arises which can be considered a potential source of error or uncertainty for the results, then it is addressed following section 6.7.

### 6.2.2 Safety Considerations

Good laboratory and scientific practices are used in the laboratory to protect against injury. Applicable LANL and/or LANL-contractor safety practices for conducting laboratory work are followed, as appropriate.

#### 6.2.3 Special Handling

Care must be taken to minimize any potential for contamination of the sample with the analyte of interest (usually chloride or $^{36}Cl$).

## 6.3 Preparatory Verification

#### 6.3.1 Hold Points

N / A

#### 6.3.2 Calibration

The analytical balance used to determine the gravel-free fraction of a soil sample (section 6.5.2) is controlled pursuant to QP-12.3. The balance used to estimate the weight of rock samples during the sieving procedure (section 6.5.3) is zeroed by the user before each use. Control of the balance is not necessary under the provisions of QP-12.3 which states that a PI may determine an instrument is exempt from control and calibration procedures. The balance is exempt from control and calibration procedures because it provides a measurement that is intended to be an approximation of the sample weight within 10%.

Similarly, the water purification system also does not require calibration because the purpose of its meter is limited to alerting the user to a failure of the system to produce deionized water, as indicated by the reading on the resistivity meter. Meter failure is indicated by a “zero” reading, at which time the meter is replaced by the vendor.

#### 6.3.3 Environmental Conditions

Measures to minimize the potential for cross-contamination by particles or sources of chloride and bromide other than those in the sample being processed are discussed in section 6.5.1. Sieving is conducted in a room with adequate ventilation. No other special environmental conditions are required.

## 6.4 Control of Samples

Sample identification and control must be sufficient to trace a sample and its derivatives from its original field location to the point of analysis, and the integrity of the sample must be safeguarded throughout analytical processes. Consequently, users must be trained to DP-103 before they can work with YMP samples and they must also follow guidelines set forth in that document for sample control.

## 6.5 Implementing Procedure

### 6.5.1 Cleaning

To minimize cross-contamination, sieves and labware are cleaned prior to processing each new soil or rock sample. Concurrent with cleaning, each sieve is also inspected to ensure that the mesh is not damaged. If the purpose of the sieving is only to determine the gravel-free fraction (section 6.5.2), after which the sample can be discarded, then cleaning of the sieves and labware is limited to wiping them free of excessive particles which might bias the sample weights. Compressed air may also be used to remove particles from the sieves.

If the purpose of the sieving is to prepare the sample for halide analyses (section 6.5.3), then additional steps must be taken with the workspace, sieves, and labware to minimize the potential for contamination of the samples. A new sample is not opened until the dust from the previous sample has settled and the work space has been wiped with a damp sponge. New labware is initially washed with detergent such as Alconox, rinsed with tap water, then rinsed three times with deionized water. Subsequent use requires rinsing three times with deionized water unless excessive sample material has contaminated a piece of labware, in which case the item is washed using the procedure for new labware. The user either wears a new pair of plastic gloves for each sample or washes his/her hands between each sample. The sieves are washed thoroughly with tap water, rinsed several times with deionized water, and finally dried completely. Compressed air may be used to speed the drying process. The sieves are visually inspected to ensure that no soil or rock particles are caught in the screens.

### 6.5.2 Determination of the Gravel-Free Fraction of a Soil Sample

Sieving a soil sample to determine the gravel-free fraction takes place after determining its moisture content (DP-90), or before leaching it for halides (DP-92). Typically, for all but the chlorine isotope analyses, sample sizes are expected to range between 50 g and 1 kg, averaging about 100-200 g. Several kg of soil may be needed for chlorine isotope analyses.

To determine the gravel-free fraction of a soil sample, the sample is oven-dried at 105°C±5°C for 24 hours, cooled in a desiccator, then weighed. The dry sample is passed through a 2 mm mesh sieve. A mortar and rubber-tipped pestle or its equivalent are used to break up those aggregates which are larger than 2 mm, taking care not to crush any individual grains. The portion retained or passed by the sieve, whichever is more convenient, is weighed. The total, retained and/or passed weights are recorded. The gravel-free weight is then either the weight of

the portion passing the 2-mm sieve, or else the difference between the weight of the total sample and the weight of the fraction retained by the 2-mm sieve. The latter method of calculation is preferred when obtaining the weight of the gravel-free portion requires transfer of very fine material to another container, thereby possible biasing the result due to loss of material during the transfer.

6.5.3 Removal of Rock Flour and Large Rock Fragments from a Rock Sample

6.5.3.1 Threshold Values for Particle Sizes

Rock flour is functionally defined in this DP as the fraction of a sample that passes through a 0.105-mm sieve. Large rock fragments are functionally defined as the fraction of the sample retained by a 1.25-cm sieve. If the PI requests that one or both of these fractions be removed from a rock sample, then sieving is done prior to leaching the sample for chloride, bromide or chlorine isotope analyses.

6.5.3.2 Sample Size Guidelines

Because of the limited capacity of the sieve and the need to keep airborne dust to a minimum, numerous individual sievings may be required until the entire sample has been sieved, depending upon the quantity of sample to be sieved. For chloride and bromide determinations, at least 1 kg may be needed. If chlorine isotopic analyses are also to be made, as much as 15 to 20 kg of material may be required. These numbers are intended only as rough guidelines.

6.5.3.3 Sieving Procedure

The PI provides guidance in determining which fractions are to be separated and hence which mesh sizes are needed. If the intent is only to remove the large rock fragments, then only the 1.25-cm sieve is needed. If the intent is to remove rock flour, then the 0.105-mm sieve is used. Other, larger mesh sizes may be stacked on top of this one to increase the efficiency of sieving large volumes of material. The selected sieve sizes are stacked onto a base, with the smallest mesh opening on the bottom, and covered with a lid in order to control dust. The sample is placed in the topmost sieve and the lid secured. The sieves may be shaken either manually or with a sieve shaker until all particles smaller than the size of interest have passed through that screen. Each sieved fraction is inspected visually to ensure that retained particles are actually larger than the mesh opening and not just aggregates of smaller particles. If particles appear to be aggregates, they can be broken up using a spatula or similar device.

6.5.3.4 Treatment of Large Rock Fragment Fraction

If collected, the large rock fragment fraction (i.e., >1.25-cm mesh size) may be either discarded, returned to storage, or crushed to smaller sizes such as described by procedures in DP-109. Whether and how to crush these fragments is at the user's discretion. If appropriate, the crushed sample is then resieved following step 6.5.3.3 to remove rock flour generated during the crushing process.

6.5.3.5 Transfer of Sample after Sieving

When sieving of a given sample is complete, the rock flour fraction (if collected) is discarded. The fractions to be retained for analysis are combined by transferring them, one at a time using a plastic scoop or gloved hand, into clean, tared or preweighed containers or weighing boats. The containers are covered or sealed after each transfer. The total sample weight is measured or calculated.

6.5.3.6 Subsequent Portions

If additional material needs to be sieved in order to ensure a sample of adequate size for analysis, then the next portion is transferred into the topmost sieve and steps 6.5.3.3 through 6.5.3.5 are continued until sufficient sample has been sieved.

6.5.3.7 Residual Material

Any unsieved sample may be returned to the original container in storage in accordance with DP-103.

## 6.6 Data Acquisition and Reduction

Weights are recorded to the nearest 0.01 gram or better. The acceptability and precision of the data are evaluated by the PI, taking into account the precision of the instruments used as documented pursuant to QP-12.3.

## 6.7 Potential Sources of Error and Uncertainty

Careful labeling of containers reduces errors due to mislabeling. The possibility of sample contamination is reduced by working in a clean environment, wiping down surfaces prior to and after sieving, and cleaning the sieves prior to sieving. The user is responsible for documenting deviations from this DP and any problems which could be considered potential sources of error or uncertainty for the results, both in accordance with QP-03.5.

## 7.0 RECORDS

Records to be generated as a result of the proper execution of this DP are entries in laboratory notebooks and in the Sample Inventory Logbook. These records are controlled by procedures QP-03.5 and DP-103. A checklist for laboratory notebook entries relevant to this DP is provided in Attachment 1.

## 8.0 ACCEPTANCE CRITERIA

The criteria that show that this procedure has been correctly implemented are the records identified in Section 7.0.

## 9.0 TRAINING

A prerequisite for this DP is to train to DP-103. This DP requires read-only training. Training of personnel to this DP is documented pursuant to QP-02.7.

## 10.0 ATTACHMENTS

Attachment 1: Checklist of Laboratory Notebook Entries (1 page)

# CHECKLIST OF LABORATORY NOTEBOOK ENTRIES

Initial descriptive information is entered in the laboratory notebook as appropriate prior to starting a technical procedure and on a continuing basis as experimental and procedure changes dictate. These entries are:

- reference to this DP, including revision number
- unique identifiers for any calibrated equipment used to conduct this DP

Additional information is entered into the laboratory notebook a minimum of once a day for each day that relevant laboratory work is done, and more often if conditions change. These entries are:

- date of entry
- problems (if any) which may pose a potential source of error or uncertainty for the results

In addition to general entries, several parameters need to be recorded in order to evaluate the resulting data. Weights are recorded to the nearest 0.01 gram or better. These entries are:

- sample processed, listed by its unique identifier (in accordance with DP-103)
- sieve mesh sizes used
- for soil, either weight of fraction retained by 2-mm sieve or else total sample weight and weight of fraction passing the 2-mm sieve
- for rock sample, combined weights of fractions retained by 0.105-mm and coarser sieves (excluding fraction retained by 1.25-cm sieve if appropriate)